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Influence of nonthermal effects of microwave and pulsed magnetic fields on radiative recombination centers in SiC/por-SiC/Er_2O_3 structures

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Abstract. In this paper, the effect of short-term nonthermal exposure to microwave radiation and pulsed magnetic field (PMF) on photoluminescence characteristics of SiC/por-SiC/Er_2O_3 structures has been considered. Analysis of the photoluminescence spectra of these structures has shown that the changes observed in the spectra under microwave and PMF exposure are explained by an increase in the migration mobility of dislocations, which in turn leads to a redistribution of recombination centers in the SiC/por-SiC/Er_2O_3 structure. The influence of PMF on the processes of redistribution of recombination centers in the SiC/por-SiC/Er_2O_3 structure has been shown more effective than microwave exposure. The mechanisms of action of both microwave radiation and PMF are considered to be those leading to increase in the migration ability of dislocations due to change in the internal state of interacting dislocations and lattice defects.

Keywords: nonthermal microwave action, pulsed magnetic field, photoluminescence, buffer porous layer, erbium oxide, silicon carbide.

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1. Introduction

Currently, use of silicon carbide-based structures in high-power, high-temperature, and high-frequency devices [1–4] is widely demanded due to high thermal, radiation, and chemical resistance of silicon carbide. These properties allow silicon carbide to be successfully applied in microwave devices, ultraviolet photodiodes, light emitting diodes (LEDs), and as an element of metal-insulator-semiconductor (MIS) structures [5].

Reducing the scale of the MIS structure elements entails use of thin oxide films of rare earth elements as dielectrics in such structures [6, 7], since such oxides are characterized by high permittivity values (so-called high-k dielectrics) [8, 9]. One of these dielectrics is erbium oxide. A disadvantage of using thin Er_2O_3 films in MIS structures is mismatch of crystal lattice parameters of silicon carbide and Er_2O_3 (a = 1.005 nm for Er_2O_3 and a = 0.491 nm for SiC [10]), which has a significant impact on the characteristics of the semiconductor/dielectric interface.

One way to minimize the negative impact of the lattice mismatch between the film and the substrate is introducing buffer layers of a porous material. For silicon carbide – oxide film structures, such a buffer layer is porous silicon carbide, which can be easily formed on a crystalline silicon substrate. At the same time, introduction of porous layers should not lead to changes in the operation characteristics of SiC/por-SiC/Er_2O_3 structures, which will largely depend on the properties of the Er_2O_3/porous layer interface. It should also be noted that porous silicon carbide itself is a potentially attractive material for LEDs, photodetectors, and sensor devices [2, 5, 11–13].

To reduce the concentration of defect states at the oxide-semiconductor interface, additional treatments such as thermal annealing, microwave radiation treatment, and magnetic field treatment are often used. In particular, it was shown in [14, 15] that microwave radiation treatment increases transparency of semiconductor/insulator structures. As shown in [16], treatment in a magnetic field also leads to a change in the number of luminescent centers and the efficiency of radiative and non-radiative recombination channels in doped semiconductors.

According to [17], the reason for the change in transparency and photoluminescence (PL) spectra of oxide film/silicon carbide structures under nonthermal microwave exposure is an increase in the migration

ability of dislocations, which in turn leads to redistribution of emission and absorption centers in these structures. At the same time, treatment in a pulsed magnetic field (PMF) also leads to depinning of dislocations and, as a consequence, to redistribution of defects or recombination centers in semiconductors [16]. The redistribution of the defects and recombination centers under the action of both microwaves and PMF takes place due to the fact that dislocations begin to move toward the interface under the action of internal mechanical stresses, entraining the Cottrell atmospheres with them. Given that both microwave radiation and PMF enhance dislocation migration, it is of interest to compare the effectiveness of short-term low-energy microwave radiation and PMF treatment on oxide film/porous silicon carbide/silicon carbide structures.

In this study, we investigated influence of low-energy short-term nonthermal microwave and PMF irradiation on redistribution of impurity-defect states in 4H-SiC/por-SiC/Er_2O_3 structures.

2. Samples and experimental methodology

Porous silicon carbide surface (4H polytype) was obtained by electrochemical etching in a 1:1 HF:C_2H_5OH solution at a current density of 10 mA/cm^2 for 10 min. The material was then treated in molten KOH at 550 °C to open the pores. In the next step, an erbium film was deposited onto the porous silicon carbide surface by thermal spraying. The porous SiC structures with the deposited erbium layers were annealed in vacuum at 800 °C for 8 min. Subsequently, a thin Er_2O_3 oxide film was formed on the por-SiC surface by rapid thermal annealing (RTA) in a dry oxygen atmosphere at 400 °C. The thickness of the obtained oxide layers determined by Auger spectroscopy [18] was ~ 100 nm.

Microwave processing was performed in the operation chamber of a magnetron at a frequency of f = 2.45 GHz and the specific power ~$4\cdot10^{-2}$ W/cm^3. The microwave exposure time was 1 s. The total energy obtained by the structure during microwave processing was $E_{mw} \approx 4\cdot10^{-2}$ J.

During PMF processing, the structure was placed inside a solenoid. The interval between pulses was 5 s, and the pulse duration was ~$3\cdot10^{-5}$ s. The solenoid coil inductance was 11.2 μH, and the specific power of a single PMF pulse was ~$2\cdot10^{-7}$ W/cm^3. The number of the pulses during PMF processing was 160. The total energy obtained by the structure during the PMF processing was $E_{imp} \approx 3.8\cdot10^{-5}$ J.

PL spectra of the structures were obtained in backscattering geometry using a Horiba Jobin Yvon T64000 spectrometer with a confocal microscope and a cooled CCD detector.

To detect PL associated with radiative recombination in the por-SiC or por-SiC/Er_2O_3 layer, Ar-Kr laser radiation with λ_{exc} = 532.0 nm was used for excitation. Such choice is caused by the fact that the energy corresponding to λ_{exc} = 532.0 nm is $h\nu_{ex}$ = 2.3 eV, which is less than the band gap width of crystalline 4H-SiC (E_g = 3.23 eV, which corresponds to λ = 384 nm) [19].

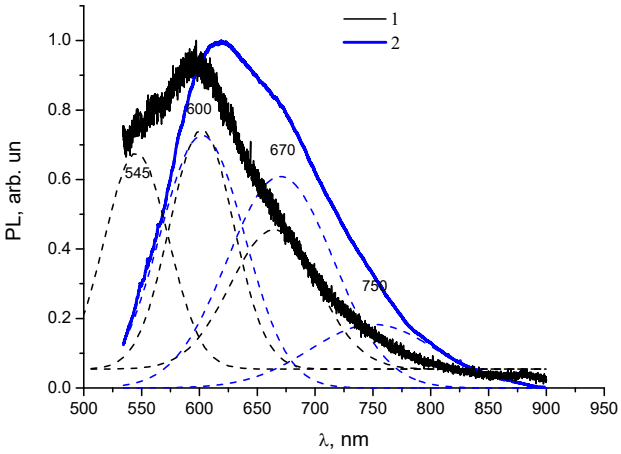

Fig. 1. Characteristic normalized PL spectra (λ_{exc} = 532.0 nm) (solid lines) together with decompositions into individual bands (dashed lines) of the initial SiC/por-SiC (*1*) and SiC/por-SiC/Er_2O_3 (*2*) structures.

3. Experimental results and discussion

Fig. 1 shows PL spectra of the initial SiC/por-SiC and SiC/por-SiC/Er_2O_3 structures normalized to the band maximum at λ_{exc} = 532.0 nm ($h\nu_{ex}$ = 2.3 eV). At the same time, it was experimentally confirmed that excitation with radiation with λ_{exc} = 532.0 nm (smaller photon energy than the band gap width of crystalline 4H-SiC, $h\nu_{ex}$ = 2.3 eV < E_g = 3.23 eV), does not induce 4H-SiC luminescence. Consequently, all the features of the PL spectra shown in Fig. 1 are caused by the por-SiC or por-SiC/Er_2O_3 layer.

As can be seen from Fig. 1, the PL bands of the SiC/por-SiC structures have large half-widths and consist of several individual PL bands with $\lambda_{max} \approx$ 545 nm, ≈600 nm and ≈670 nm. The band with $\lambda_{max} \approx$ 545 nm is associated with carbon fluoride oxide [20], which can be formed during electrochemical etching of the crystalline SiC substrate in HF:C_2H_5OH solution [21]. The band with $\lambda_{max} \approx$ 600 nm is probably caused by presence of cubic silicon carbide (3C-SiC) microcrystallites in por-SiC [22, 23]. The low-energy PL peak with $\lambda_{max} \approx$ 670 nm can be explained by radiative recombination through some localized states (shallow traps) or donor-acceptor-pairs (DAP) [24].

It should be noted that, according to [1, 19, 25, 26], appearance of luminescence of por-SiC upon excitation by radiation with an energy lower than the bandgap width of the initial Si carbide polytype (6H-SiC, E_g = 3.02 eV, or 4H-SiC, E_g = 3.23 eV), is a characteristic feature of por-SiC luminescence. As was shown in [1, 26], another feature of the PL from por-SiC is that PL spectra of por-SiC obtained from different SiC polytypes by anodic etching have practically the same spectral shapes. In the literature, this is usually associated with appearance of 3C-SiC crystallites in the porous layer [27] or with impurity-defect states formed on the por-SiC surface as a result of chemical reactions that accompany etching of the initial crystalline silicon carbide surface [1, 24, 27].

Comparative analysis of the transmission spectra of the 4H-SiC crystalline substrate and the SiC/por-SiC structures obtained by anodic etching of 4H-SiC substrate showed no shift in the position of the absorption edge for the SiC/por-SiC structure relative to that for the 4H-SiC substrate [25].

This allowed us to conclude that appearance of the PL from the por-SiC at an excitation radiation energy $h\nu_{ex} \le E_g$ (4H-SiC) is not associated with formation of an additional 3C-SiC layer in the por-SiC, since the 3C-SiC layer should shift the absorption edge of the SiC/por-SiC structure due to the smaller band gap of the cubic Si carbide polytype [21, 28]. Moreover, analysis of the Raman spectra of 4H-SiC, SiC/por-SiC and SiC/por-SiC/Er_2O_3 structures carried out in [19] showed that 3C-SiC phase is present in these structures only as structural defects, which is characteristic of all silicon carbide polytypes.

Therefore, PL from the por-SiC in 4H-SiC, SiC/por-SiC and SiC/por-SiC/Er_2O_3 structures at the excitation radiation energy $h\nu_{ex} \le E_g$ is mainly caused by emission centers formed by impurity atoms and surface defects that appear during anodic etching of the crystalline SiC substrate and subsequent processing that opens pores [19, 25]. Since the process of anodic etching during the formation of pores in silicon carbide leads to destruction of Si–C bonds [19, 25], complex compounds (such as oxides and siloxenes) and Si–H or C–H bonds, as well as C–N (nitrogen is an uncontrolled impurity in SiC) form in the porous SiC layer [1]. At the same time, individual radiative recombination centers in por-SiC can be associated with structural defects induced by stacking faults, *i.e.* 3C-SiC clusters [19, 25].

When an Er_2O_3 film is formed on a por-SiC layer, a change in the PL spectrum similar to the case of PL excitation from the SiC/por-SiC/Er_2O_3 structures by radiation with λ_{exc} = 488.0 nm [19] is observed (Fig. 1). Hence, formation of the Er_2O_3 film leads to quenching of the band with $\lambda_{max} \approx$ 545 nm, and appearance of an additional band with $\lambda_{max} \approx$ 750 nm. The relative intensity of the band with $\lambda_{max} \approx$ 670 nm increases and the relative intensity of the band with $\lambda_{max} \approx$ 600 nm remains practically unchanged (Fig. 1). The appearance of the band with $\lambda_{max} \approx$ 750 nm in the PL spectrum of the SiC/por-SiC/Er_2O_3 structure upon deposition of an oxide film can be associated with radiative recombination at oxygen-related defects that appear as a result of additional oxidation of the porous surface of silicon carbide [24].

Fig. 2 shows PL spectra of the initial SiC/por-SiC/Er_2O_3 structures and after microwave radiation and PMF exposure, normalized to the band maximum intensity, together with the spectra decomposition into individual bands. The excitation radiation energy was $h\nu_{ex}$ = 2.3 eV < E_g (4H-SiC) = 3.23 eV. As can be seen from the inset in Fig. 2, microwave radiation exposure leads to an insignificant increase in the intensity of the band with $\lambda_{max} \approx$ 670 nm. Weak changes in the PL spectra of the SiC/por-SiC/Er_2O_3 structures after microwave radiation exposure as compared to the PL spectra of the initial structures can be explained by the very short exposure time of about 1 s.

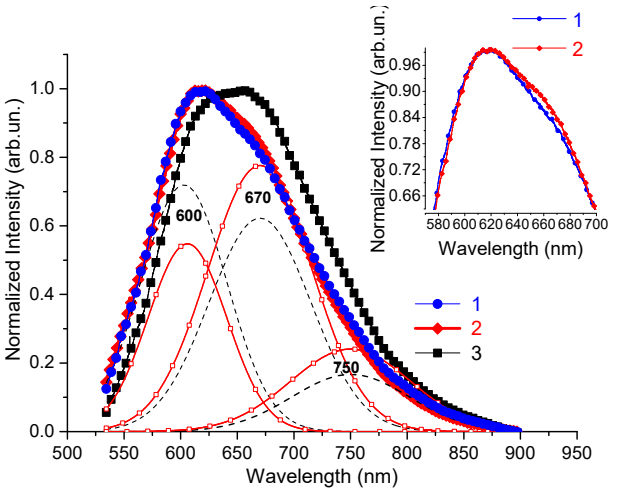

Fig. 2. Normalized to the maximum intensity PL spectra (λ_{exc} = 532 nm, $h\nu_{ex}$ = 2.3 eV < E_g (4H-SiC) = 3.23 eV) and decompositions into elementary bands for the SiC/por-SiC/Er_2O_3 structures before (*1*) and after exposure to microwave radiation (*2*) and PMF (*3*). The inset shows normalized PL spectra (λ_{exc} = 532 nm, $h\nu_{ex}$ = 2.3 eV < E_g (4H-SiC) = 3.23 eV) of the SiC/por-SiC/Er_2O_3 structures before (*1*) and after exposure to microwave radiation (*2*).

As shown in [19], microwave irradiation for 5 s leads to a more significant change in the intensity ratio of individual bands constituting integrated PL spectra of SiC/por-SiC/Er_2O_3 structures and to quenching of the band with $\lambda_{max} \approx$ 750 nm, which results in a short-wave shift of the integrated PL maximum and a decrease in the PL spectrum half-width. The authors of [19] attribute this effect to redistribution of radiative recombination centers caused by detachment of dislocations from stoppers as well as to changes in the concentrations of Si–H, C–H or C–N bonds in the por-SiC buffer layer.

As can be seen from Fig. 2, the effect of PMF on the SiC/por-SiC/Er_2O_3 structure consists in a slight broadening and a shift of the maximum of the integrated PL spectrum toward longer wavelengths due to a change in the intensities of elementary PL bands. The relative intensities of the bands with $\lambda_{max} \approx$ 670 nm and $\approx$ 750 nm increase after the PMF exposure, and the relative intensity of the band with $\lambda_{max} \approx$ 600 nm decreases. Note that the total energy obtained by the structure during the PMF treatment is 10^3 less than the total energy obtained by the structure during microwave radiation treatment. However, as can be seen from Fig. 2, the effect of PMF on redistribution of recombination centers in the SiC/por-SiC/Er_2O_3 structure is more pronounced than that of microwave radiation exposure.

It is well known that PMF treatment usually leads to detachment of dislocations from stoppers followed by their movement in the structure under the action of mechanical stresses existing in this structure [28, 29]. Such processes are called the magnetoplastic effect in the literature [28, 29]. Therefore, we associate the changes observed in the PL spectra of the SiC/por-SiC/Er_2O_3 structures after the PMF treatment (Fig. 2) with the processes caused by the magnetoplastic effect.

The magnetoplastic effect is associated with a change in the internal state of interacting dislocations and lattice defects or impurity ions, primarily paramagnetic centers in non-magnetic crystals under the action of external magnetic field [28, 29].

According to [28, 29], unsaturated orbitals with unpaired spins are present in the dislocation cores in various crystals. Interaction of such orbitals with paramagnetic impurities leads to formation of radical pairs (RPs) in singlet (*S*) or triplet (*T*) states. Typically, the *S*-state is at a lower energy level. Therefore, detachment of a dislocation from an impurity is defined by the RP rupture energy in the *S*-state. In a magnetic field, intercombination transitions between the *S*- and *T*-states become possible, leading to an increase in the population of the *T*-states of RPs, which have significantly lower rupture energies, which in turn leads to an increase in the dislocation mobility. Transitions between the states with different multiplicities are possible only at the stage of RP emergence, when the exchange interaction is weak and the term levels are practically identical.

In this case, the *S*–*T* transitions are a resonant process [28, 29]. Intercombination transitions occur either due to the difference in the Zeeman frequencies of electrons in a pair (Δg mechanism) or due to hyperfine interaction of electrons with magnetic nuclei. In this case, structural defects can act as electrically active centers, which have the character of singly or multiply charged ionized donors and acceptors [28, 29]. Note that under the influence of both microwaves and PMFs, the main effect on the structure is caused by activation of resonant processes in it. However, in the case of microwaves, the resonant effect is due to the microwave resonance with oscillations of the stopper-dislocation system, while in the case of PMFs, singlet-triplet transitions in the paramagnetic center act as a resonant process.

In silicon carbide, uncontrolled nitrogen impurity often acts as a paramagnetic defect, which can occupy nonequivalent positions with cubic and hexagonal symmetry in a lattice [30–34]. Depending on the polytype, the number of such positions can vary from two cubic and one hexagonal in 6H-SiC to five nonequivalent positions in 15R-SiC. Here, *N* substitutes for five nonequivalent positions: two hexagonal and three quasi-cubic ones [30–34]. It was also shown in [30–34] that nitrogen atoms can substitute both Si and C atoms in the silicon carbide lattice, forming two types of energy schemes for nitrogen donor levels. Moreover, two types of nitrogen donors are assumed in presence of randomly distributed local stresses in silicon carbide. One type replaces strained sites and another type replaces unstrained sites, which leads to appearance of additional paramagnetic centers [30–34].

As shown in [30–34], even in the absence of a directed external influence, random fluctuations of the magnetic field cause spin diffusion due to spin–spin interaction between *N* donors and free electrons, which leads to random excitation of the spin system [30–34]. Due to high sensitivity of the dislocation – paramagnetic center spin system, influence of PMF initiates spin-dependent transitions between different levels of nitrogen impurity, which entails changes in the spin state in the dislocation – paramagnetic center system. In turn, the mentioned change in the spin state leads to a short-term transition of the dislocation – paramagnetic center system from the bonding to the antibonding state, which radically changes the system configuration, causing detachment of dislocations from local defects [28, 29]. Subsequent motion of the dislocations occurs under the action of internal stress fields in the crystal structure.

It should be noted that the motion of the dislocations and associated Cottrell atmospheres under the action of PMF and in the case of microwave irradiation [17] can lead to redistribution of impurities and defects in the SiC/por-SiC/Er_2O_3 structure. This results in a change in the nature of the interimpurity interaction between the luminescence centers [35, 36] and a change in the symmetry of the nearest environment of the impurity atoms in silicon carbide.

As can be seen from the presented results, PMF treatment is more effective, since the redistribution of the recombination centers of the SiC/por-SiC/Er_2O_3 structure by the PMF treatment requires significantly lower total energy. The total energy obtained by the structure during the PMF treatment is 10^3 times lower than the total energy obtained by the structure during the microwave radiation treatment. The higher efficiency of the PMF treatment compared to the microwave radiation treatment can be explained by the fact that although both treatments lead to redistribution of recombination centers in the structure due to resonant interaction, this resonant effect has different natures.

In the case of microwave exposure, an external force causes resonant oscillations of the stopper–dislocation system. The dislocation detachment from the stopper is caused by a resonant increase in the oscillation amplitude. Moreover, under microwave radiation exposure, part of the energy is transferred to the crystal lattice of the entire structure. It is possible that a part of the microwave radiation energy may be spent, among other things, on changing the concentrations of Si–H, C–H, or C–N bonds in the por-SiC buffer layer and further oxidation of silicon in this layer [19], since, according to [37], microwaves can also serve as a catalyst for chemical reactions.

In the PMF case, the resonant effect that leads to dislocation detachment from stoppers is the spin conversion mechanism, which is accompanied by spin-dependent transition of the dislocation – paramagnetic center system from the bonding state to the antibonding state due to intersystem crossings between the *S*- and *T*-states of the paramagnetic center. In this case, almost all of the IMP energy is spent on initiating spin-dependent transitions in the dislocation – paramagnetic center system.

4. Conclusions

In conclusion, both PMF and microwave irradiation lead to redistribution of recombination centers in SiC/por-SiC/Er_2O_3 structures. At this, PMF irradiation may be considered more effective. Different efficiencies of the microwave and PMF irradiation are caused by the fact that in the former case, resonant processes lead to excitation of the oscillatory subsystem of the entire structure, whereas in the case of PMF irradiation, the resonant process is the spin conversion mechanism, which can occur only in localized regions of the structure.

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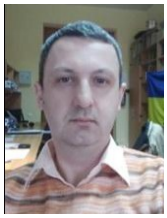

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Атермічний вплив НВЧ та імпульсного магнітного поля на центри випромінювальної рекомбінації у структурах SiC/por-SiC/Er_2O_3

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Анотація. У цій роботі розглянуто короткочасний атермічний вплив надвисокочастотного (НВЧ) випромінювання та імпульсного магнітного поля (ІМП) на фотолюмінесцентні характеристики структур SiC/por-SiC/Er_2O_3. Аналіз спектрів фотолюмінесценції таких структур показав, що зміни, що спостерігаються у спектрах при впливі НВЧ та ІМП, пояснюються збільшенням міграційної рухливості дислокацій, що, у свою чергу, приводить до перерозподілу центрів рекомбінації у структурі SiC/por-SiC/Er_2O_3. Показано, що вплив імпульсного магнітного поля на процеси перерозподілу центрів рекомбінації у структурі SiC/por-SiC/Er_2O_3 є більш ефективним, ніж НВЧ вплив. Як механізми впливу як НВЧ, так і ІМП розглядаються механізми, що приводять до збільшення міграційної здатності дислокацій за рахунок зміни внутрішнього стану взаємодіючих між собою дислокацій і дефектів ґратки.

Ключові слова: атермічна мікрохвильова дія, імпульсне магнітне поле, фотолюмінесценція, буферний пористий шар, оксид ербію, карбід кремнію.